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PATENT ABSTRACTS OF JAPAN

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(54) PREPARATION OF TETRAMETHYLUREA

(57)Abstract:
PURPOSE: To prepare the titled compound useful as an intermediate of pharmaceuticals, etc., efficiently, with easy extraction procedure, by keeping a reaction mixture of dimethylamine with phosgene at specific temperature and condition in the presence of a water-insoluble organic solvent, and then separating the mixture into an oil phase and an aqueous phase.
CONSTITUTION: Dimethylamine is made to react with phosgene in an aqueous medium in the presence of an alkali (e.g. NaOH, KOH, etc.) to obtain tetramethylurea. In the above process, the reaction mixture is kept at ≥40°C (preferably under atmospheric pressure, and in that case, usually at 40W100°C, preferably 50W90°C) under the condition to allow the water in the reaction mixture to present as a liquid phase (e.g. for 3W120min) in the presence of 0.6W 6pts.wt., based on 1pt.wt. of the tetramethylurea in the reaction mixture, of a water-insoluble organic solvent (e.g. n-heptane, n-hexane, chloroform, etc.) and the resultant mixture is separated into the oil phase containing tetramethylurea and the aqueous phase containing an alkali metal chloride. Tetramethylurea is separated from the oil phase.

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101: 90441a **Tetramethylurea.** Mitsubishi Chemical Industries Co., Ltd. **Jpn. Kokai Tokkyo Koho JP 59 31,752 [84 31,752]** (Cl. C07C127/15), 20 Feb 1984, Appl. 82/141,970, 16 Aug 1982; 5 pp. $Me_2NCONMe_2$ (I) was prepd. by treating Me_2NH with $COCl_2$ in aq. NaOH or KOH and extd. with org. solvents at > 40°. Thus, 124 g 50% aq. Me_2NH soln. was treated with $COCl_2$ (75 g/h) in the presence of 264 g 25% aq. NaOH at 0–10° for 1 h and the reaction mixt. was stirred with PhCl at 80° 30 min to give I (from org. layer) with 87% recovery rate compared with 54% at 25°.

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Tetramethyl urea prepn. - by reacting dimethylamine with phosgene in aq. medium in presence of alkali and keeping in presence of water-immiscible organic solvent

C84-034068

The reaction mixt. is kept in the presence of water-immiscible organic solvent at 40 deg.C or more (50-90 deg.C) under conditions that water in the reaction mixt. exists in the liquid phase. Tetramethyl-urea-contg. oil layer and alkali chloride-contg. aq. layer are sepd. from each other.

The amt. of organic solvent is pref. 0.4-6 pts. wt. based on tetramethyl-urea in reaction mixt. Phosgene is used in amt. of 0.5-2 pts. mol based on dimethylamine, and alkali in amt. of 0.8-6 equivs.

Extraction efficiency of tetramethyl-urea is high (e.g. 87%).

(5pp Dwg.No.0/0)